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METHODOLOGICAL APPROACH TO OPTIMIZING THE PROPERTIES OF MOLDING PASTES FOR EXTRUSION (REVIEW)

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Data characterizing the properties of molding pastes for extrusion of thin-wall articles with complex profiles are presented. It is proposed that parameters such as the deformation ratio, the relaxation period, the power required to break down the coagulation structure, and the flow index be used to characterize the molding properties of the pastes. Pastes with developed plastic properties and strong coagulation structure are recommended for extrusion.

Key words: molding pastes, extrusion, deformation ratio, relaxation period, strength of coagulation structure, flow index.

Extrusion molding of highly concentrated suspensions (molding pastes) is essentially a process where paste flows in an extruder through a die channel. Three criteria in combination determine the possibility of obtaining an extrudate with the prescribed shape [1].

In the first place, there is the geometry of the extrudate. For example, to obtain cylinders (simplest shape) the molding paste passes through a section with a large diameter and then acquires, through a conical transition, a prescribed shape by flowing out through a die with a smaller diameter. For ring extrusion there are additional obstacles in the form of, for example, cores in the flow path of the paste. However, the extrusion of blocks of a honeycomb structure involves an even more complex flow regime change. First there is a change from one diameter to another. Then the total flow is divided into many flows. Finally, these flows transition from a cylindrical to a slot shape and merge into a monolith. Thus, the flow of the molding paste in the extruder is very complicated. As is well known from hydrodynamics, a change in the cross section of a channel causes the fluid flow regime to break down [2]. These phenomena can have a large effect on the quality of the extrudate obtained [3 – 6].

The second factor which affects extrusion is the material of the extruder itself, first and foremost, the die material. The effect of this factor is manifested through the cohesion-adhesion interaction in the material — molding paste system. Thus, in [7, 8] this phenomenon is evaluated by means of the coefficient of external friction in the molding paste — die material pair. It has been concluded on the basis of these studies that to mold successfully articles with a prescribed shape the ratio of the cohesion of the molding paste to the die material must be as low as possible.

The third factor determining the success of the extrusion the are physical-chemical (specifically, structural-mechanical and rheological) properties of the molding paste. This is probably the most important factor [1, 9].

A quite large collection of the most diverse parameters is required to give a complete description of the properties of molding pastes [1]. As a rule, these parameters are directly related with other characteristics of molding pastes. The experience gained in working with the most diverse systems shows that often it is important to know not the absolute value of a particular quantity but rather its ratio to other parameters of a molding paste [1, 9]. Thus, it is necessary to determine which parameters of a molding paste can be singled out as criteria for moldability and which values of these criteria are determined by the prescribed geometry. It can be asserted a priori that the optimal values will differ over a wide range for obtaining, for example, cylindrical granules and articles with a complex profile. In the latter case the requirements for molding pastes will be much more stringent [1, 9].

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What are the requirements which molding pastes must satisfy? Here the following criteria can be singled out [1]:

plastic deformations must develop in molding pastes to the necessary degree; in other words, the paste must assume the prescribed shape precisely during extrusion through the die;

the coagulation structure of molding pastes must be strong enough so that the rheological properties are preserved in an acceptable range directly in the molding machine under the action of high shear stresses;

after leaving the die the extrudate must retain the prescribed shape and be suitable for subsequent technological operations (transport, dry-curing, drying, and so on);

the article formed must not have any macrodefects that lower its mechanical strength and degrade the commercial appearance of the finished products.

The requirements listed above are closely interrelated and at the same time contradictory. This shows the complexity and multifacetted nature of the problem of optimizing the properties of molding pastes.

Methods for measuring the properties of molding pastes for extrusion are discussed in detail in [10], where the division of these properties into structural-mechanical and rheological is presented. All measurements of the structural-mechanical rheological properties of the molding pastes, which will be discussed below, were measured with the optimal moisture content for molding [1].

An indicator that ties the molding pressure to the parameters that characterize the properties of molding pastes is needed to monitor the molding process in extruders of articles from highly filled molding pastes. Such an indicator of paste moldability with extrusion is proposed in the works of V. S. Fadeeva [11, 12] and is called the moldability coefficient. This coefficient is determined from tests performed on a stamp with a constant cross section [1, 10 – 12]. The optimal value of the moldability coefficient is ± 0.5 [11, 12]. However, it is shown in [1, 13, 14] that this criterion is not universal.

The ratio of different types of deformations serves as another suitability criterion for molding pastes. It should be noted that the indicator is one of the most popular indicators for different authors, as is indicated by the large number of works devoted to molding, where this criterion is used. However, up to now there is no unanimity as to which deformation ratio is optimal.

Thus, molding pastes belonging to structural-mechanical types I and especially II, i.e., with slow elastic deformations predominating, are recommended in [15, 16] for extrusion. The predominant development of fast elastic deformations (structural-mechanical types 0 and III) results in brittle fracture of the extrudate, and pastes with substantial development of plastic deformations (structural-mechanical types IV and V) manifest, in the opinion of these authors, a tendency toward plastic breakage, specifically, cord formation [16].

The choice of predominant development of slow elastic deformations as optimal is made on the basis of an investigation of the structural-mechanical properties of ceramic pastes, according to which the optimal values of the elasticity ($\lambda = 0.6 - 0.65$), plasticity ($Ps = (2.0 - 2.5) \times 10^{-6}\ sec^{-1}$), and relaxation period ($\Theta = 1200 - 1400$ sec) were determined [16]. It was established in the same work that pastes belonging precisely to structural-mechanical types I and II meet these requirements.

The same values of the elasticity and plasticity as those indicated are recommended in [17] as optimal. As for the deformation ratio, the authors single out the pastes suitable for extrusion of honeycomb blocks, which are of type 0 or I.

In [18], for successful extrusion preference is given to molding pastes with uniform development of all types of deformations and no structural-mechanical types are concretely singled out.

At the same time all authors arrived at a unanimous opinion:

1) it is inadmissible for elastic deformations which result in brittle breakage of the extrudate to predominate;

2) all types of deformations must be developed to the proper degree, i.e., the fraction of deformations of any one type should not exceed 70%;

3) the structural-mechanical properties alone are insufficient to give a confident characterization of the suitability of a paste for extrusion with a prescribed geometric shape; a comprehensive investigation must be made using, specifically, the rheological parameters, as is indicated in [1, 9, 10, 19] and [17].

Work experience has shown that the blocks of a honeycomb structure are most easily formed from ceramic pastes, specifically, ultraporcelain pastes [1, 9]. For this reason, a given paste can be regarded as a standard and its properties can be considered as optimal for the formation of articles with a complicated profile and thin walls. Measurements of the structural-mechanical properties of this paste have shown that predominately plastic deformations are observed to develop in it and it is a structure-mechanical type IV paste (Fig. 1). It is also noted in [20] that structure-mechanical type IV or V pastes are best for forming blocks of a honeycomb structure. Experience in working with different pastes also shows that the best pastes for forming, for example, honeycomb blocks are pastes where plastic deformations develop predominately 40 – 70% and which are of structure-mechanical types IV or V. The authors of [17] indicate that a block structure can be formed from pastes based on iron oxide and aluminum hydroxides using systems in which slow elastic deformations predominate. All this has made it possible to determine the region of deformation ratios for a paste to be used to form blocks of a honeycomb structure. In Fig. 1 this region marked by hatching. It is clearly seen in the figure that all samples presented fall within the region singled out. However, by no means is every paste suitable for extruding articles with complex profiles and thin walls. For

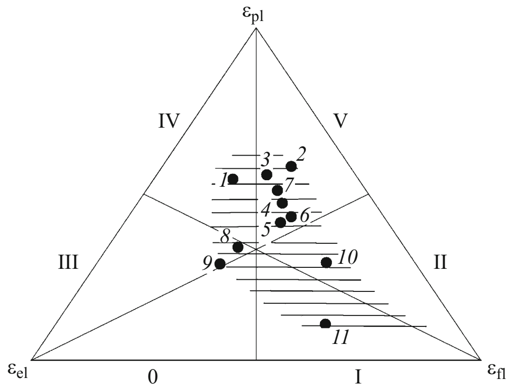

Fig. 1. Diagram of the development of deformations in molding pastes: *1*) ultraporcelain; *2*) Al_2O_3 – paraffin; *3*) Al_2O_3 – PVC; *4*) TiO_2 – PVC; *5*) TiO_2 – clay – PEO; *6*) TiO_2 – clay – PVC; *7*) graphite – CMC; *8*) aluminum titanate – PEO; *9*) aluminum titanate – CMC; *10*) Fe_2O_3 – AlOOH; *11*) Fe_2O_3 – AlOOH – $Al(OH)_3$.

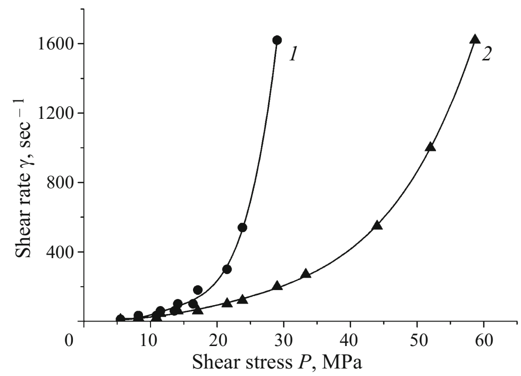

Fig. 2. Rheological curves of molding pastes based on Al_2O_3 – paraffin: *1*) comminuted Al_2O_3 ($\Delta N = 12.8$ MW/m^3); *2*) Al_2O_3, comminuted together with paraffin ($\Delta N = 24.9$ MW/m^3).

example, a honeycomb block can be obtained from an ultraporcelain paste, pastes based on Al_2O_3 using paraffin, and pastes based on a mixture of TiO_2 and clay, where polyethylene oxide was used as a plasticizer, and pastes made from aluminum titanate and iron hydroxide.

For extrusion of cylindrical granules, the region of optimal development of deformations will be much wider and, aside from that indicated in Fig. 1, will extend to all structure-mechanical types.

Therefore, a definite deformation ratio is necessary but insufficient condition for the suitability characteristics of molding pastes.

The criteria according to which the strength of a coagulation structure can be evaluated are the total power N expended on the flow and the power ΔN expended on the destruction of the coagulation structure. These parameters are determined from the complete rheological curves of the pastes [10]. The role of the values of N and ΔN in the extrusion process is as follows.

In the first place, just as the admissible shear stress, these parameters determine the strength of the green molded article after the paste has left the die. This is especially important for extrudates with thin walls, where the wall thickness can be 0.1 – 2 mm. If for cylindrical granules or rings with wall thickness of the order of 4 – 5 mm inadequate strength of the green articles results only in slight crumpling, then for thin-wall extrudates this will result in strong deformation under the action of the paste itself immediately after the extrudates exit from the die, and there is no longer any need to talk about successful molding.

In the second place, comparatively high values of N and ΔN are necessary for a molding paste directly during the extrusion process. Here is why. A die has a quite high hydraulic resistance, and the more complicated the shape of the extrudate, the higher the resistance of the die. An external pressure must be applied to overcome this resistance to the molding paste; this pressure is created in extrudates by an augur or a plunger. For pastes that are Newtonian liquids it is known that as the shear stress increases, the viscosity can decrease by several orders of magnitude. However, if the coagulation structure of the molding paste is too weak, then in the process of extrusion strong liquefaction can be observed as it passes through the die channels, i.e., the transition of the flow into a regime with a structure that is breaking down energetically. After leave the molding machine there is not enough time for the paste to restore its initial properties thixotropically. However, a stronger coagulation structure makes it possible to work under more intense external actions.

We shall illustrate this with an example. Figure 2 shows the flow curves of molding pastes based on Al_2O_3 which have the identical composition but are prepared by different methods, as a result of which the strength ΔN of the coagulation structure differs by a factor of 2. The extrusion molding process proceeds in a regime with a practically intact structure (the section of the curve which is flatter toward the abscissa). It is evident from the data in Fig. 2 that this regime of the paste flow with a large value of ΔN is much wider. Thus, this section is of the order of 20 kPa for the first sample and 40 kPa for the second one. Therefore, higher shear stresses can be applied to the second paste during extrusion without risk of completely destroying its coagulation structure.

A strong coagulation structure will involve in an obvious manner an increase of the energy consumption for conducting the extrusion process, since the system will possess a very high viscosity. The final objective must be clear. If the problem is to transport a suspension, then, of course, it is desirable to have a coagulation structure with the lowest possible strength. For extrusion technology the objective is to obtain an article with a prescribed shape. In this case energy conservation can be sacrificed, but the required shape must be obtained.

In discussing the question of the optimal deformation ratio, the paste region for obtaining articles with complex profiles was singled out (see Fig. 1). All samples presented in

TABLE 1. Structural-Mechanical and Rheological Properties of Molding Pastes (Sample Numbers Correspond to Fig. 1)

Sample No.	Sample	Rheological properties				Structural-mechanical properties			Structural-mechanical type
		total power for flow N, MW/m^3	power for destroying the coagulation structure ΔN, MW/m^3	consistency constant η_0,** Pa · sec^n	flow index n**	plasticity Ps × 10^6, sec^{-1}	elasticity λ	relaxation period Θ, sec	
1	Ultraporcelain	57	46	130	0.26	1.0	0.2	900	IV
2	Al_2O_3, paraffin	41	25	125	0.29	0.6	0.8	700	V
3	Al_2O_3, PVC	14	10	110	0.35	2.0	0.5	500	V
4	TiO_2, PVC	8	4	16	0.63	3.3	0.6	1050	V
5	TiO_2, clay, PEO	24	16	31	0.38	1.0	0.6	1350	V
6	TiO_2, clay, PVC	48	18	53	0.42	3.2	0.6	1400	V
7	Graphite, CMC	5	2	20	0.46	4.3	0.4	2000	V
8	Aluminum titanate, PEO	18.6	14.1	110	0.29	3.4	0.4	1815	III
9	Aluminum titanate, CMC	17.9	13.3	105	0.30	3.2	0.4	2510	III
10*	Fe_2O_3, AlOOH	–	–	–	–	2.3	0.7	8110	I
11*	Fe_2O_3, AlOOH, $Al(OH)_3$	–	–	–	–	5.7	0.7	2390	II

* Data of [17].

** The values of η_0 and n were calculated from Stokes equation $\eta = \eta_0 \gamma^{n-1}$, where η is the effective viscosity, Pa · sec, and γ is the shear rate, sec^{-1}.

this figure are in this region. However, by no means all pastes are suitable for molding articles with a complicated shape. For example, a paste based on graphite with carboxymethyl cellulose has a total flow power N and power for breaking down the coagulation structure ΔN 4.6 and 2.0 MW/cm^3, respectively (see Table 1). When such a paste is used to extrude an article with a complex shape, in the process of passing though the die channels under the action of an external shear stress the article passes into a flow regime whose structure has been practically destroyed and literally flows out of the die. Molding pastes based on Al_2O_3 and TiO_2 with polyvinyl alcohol as the binder likewise have values of N and ΔN that are too high, which results in phenomena similar to graphite-based pastes but less strongly expressed. We note that satisfactory quality is attained when cylindrical granules and rings are extruded from the pastes mentioned. This shows that the strength requirements for coagulation structure likewise will depend on the article shape that must be obtained.

Of the systems presented in Table 1, extrudates with a complex profile were obtained from samples 1, 2, 8, 9, 10, and 11. In [1, 9], for extrusion of blocks of a honeycomb structure the total power expended on the flow and the power expended on breaking down the coagulation structure were determined to be at least 20 and 15 MW/m^3, respectively. For extrusion of simpler shapes these minimum values will be much lower.

We shall now examine the effect of structural-mechanical characteristics such as the plasticity, elasticity, and relaxation period on the extrusion process. For this we turn once again to Table 1.

According to the data presented, the plasticity values vary from 0.6×10^{-6} to 4.3×10^{-6} sec^{-1}. In addition, the plasticity can reach $(10 - 11) \times 10^{-6}$ sec^{-1}, and blocks of a honeycomb structure can be obtained from such pastes. Thus, the static plasticity cannot serve as a unique criterion for suitability of a molding paste for extrusion. The physical meaning of plasticity also proves this once again — the rate of development of plastic deformations [1]. How rapidly plastic deformations develop in time is of no importance for obtaining a high-quality extrudate; the main factor is the fraction of the plastic deformations, while their rate of development will be reflected only on the productivity of the extruder.

Another structural-mechanical characteristic is the elasticity. For the samples presented in Table 1 it likewise varies over a quite wide range 0.2 – 0.8. Turning to the physical meaning of this quantity (fraction of the slow elasticity in the total elasticity, neglecting the plastic properties of the system), it becomes clear that this quantity likewise cannot be used as a criterion for paste suitability for extrusion. This is confirmed by the suitability of pastes with low elasticity (sample 1) and high elasticity (samples 10, 11) for extrusion of blocks of a honeycomb structure. That is, the ratio of only the fast and slow elastic deformations cannot determine the suitability of pastes for molding.

The relaxation period Θ should be especially singled out among the structural-mechanical characteristics. Interest in this parameter is entirely consistent, since relaxation phenomena play an important role in molding systems such as pastes for extrusion of catalysts and sorbents [21]. Generally speaking, from the standpoint of rheology there is no funda-

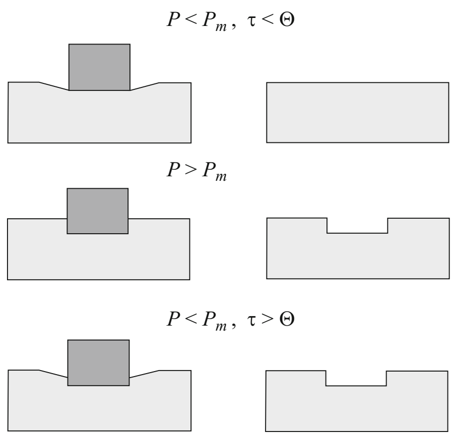

Fig. 3. Physical meaning of the relaxation period: P) external load; P_m) maximum shear stress; Θ) relaxation period.

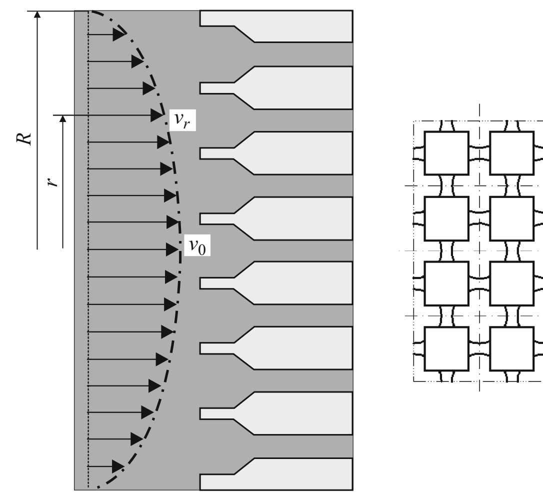

Fig. 4. Diagram of a die for extrusion of blocks of a honeycomb structure: R) channel radius; r) running distance from the flow center; v_0) flow velocity at channel center; v_r) flow velocity at distance r from channel center.

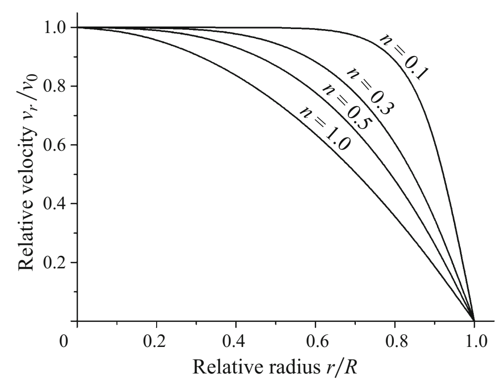

Fig. 5. Radial distribution of the relative flow velocity in a die channel as a function of the flow index (see Fig. 4 for explanation).

mental difference between a liquid and solid body. All depends on the magnitude of the load and the time over which it is applied. We shall clarify this using some examples (Fig. 3). If the applied load is less than the plastic strength of the material and the duration of its application is less than the relaxation period, then after the load is removed the deformations will vanish completely. If the load is greater than the plastic strength, then plastic deformations will remain. However, if the load is less than the plastic strength but the application time of the load is quite long, then some deformations will vanish but the remaining ones will convert to plastic deformations and remain.

In [1, 9, 20 – 22] it is indicated that during extrusion the flow in a die channel must be a steady-state flow. For this it is desirable that the relaxation period be less than the passage time of the paste through a die channel, i.e., one of the following conditions must be satisfied: either the relaxation period is short or the molding channel of the die is long. Here it must be noted that increasing the length of the molding channel will increase the hydraulic resistance of the die. This will cause the external load applied to the molding paste to increase, which can result in a transition from a flow regime with virtually intact structure into a regime with rapidly failing structure. In the process the viscosity of the molding paste decreases by several orders of magnitude, and after exiting the die channels there may not be enough time for the initial properties to be restored thixotropically, which will result in deformation of the extrudate. The conclusions drawn in [21, 23, 24] show that for large values of the relaxation period the formation of irregular defects when the extrudate leaves a channel is observed. Thus, pastes with short relaxation periods (500 – 2000) are preferable.

One of the parameters having the strongest effect on the flow process is the flow index n [1, 9, 14, 19]. Figure 4 shows the flow velocity distribution along a channel; this distribution is of the Poiseuille type. We shall analyze the flow of the paste in the extruder during the formation of the blocks of a honeycomb structure. For simplicity, we shall examine a plunger molder, since the flow in it is not made any more complicated by the large axial and radial mixing in the flow which is observed in screw extruders. The feeding part of the dies comprises a system of individual channels, located at different distances from the center of the flow (see Fig. 4). Thus, the flow rate in a concrete channel located at a distance r from the center will be determined by the velocity v_r of the paste on the section under study. It is natural to assume that this will also affect the velocity distribution in the molding channels of the dies. Therefore low values of the flow index are more advantageous for molding articles with complex profiles, since the flow velocity of the paste at the center and periphery of a die will differ by a small amount (Fig. 5). In the opinion of the authors, the most acceptable value of the flow index n is no greater than 0.3. However, when molding honeycomb blocks from pastes with a much larger index, for example, from pastes based on TiO_2 and clays to which polyvinyl alcohol was added ($n = 0.42$) (see

Table 1), the "opening flower" effect is observed; this effect is probably due precisely to the large difference of the flow velocities between the center and periphery of the dies.

In summary, it is impossible to draw any conclusions as to whether or not a paste is suitable for extrusion from measurements of the rheological properties of the molding pastes performed by means of only one method; a complex of studies is necessarily required. The main criteria for judging the suitability of pastes for extrusion are:

– *structural-mechanical*: deformation ratio, relaxation period;

– *rheological*: power expended on breaking down the coagulation structure, flow index.

The values of the parameters which must be taken into account are determined by the required shape of the article.

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